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28 September 2000.

Commissioner of Patents and Trademarks
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Dear Sir: **RE: New Patent Application My file no. UWO3**
 Title: Continuous Ion Exchange using a Liquid-Solid Circulating
 Fluidized Bed
 Inventor(s): BASSI et al.

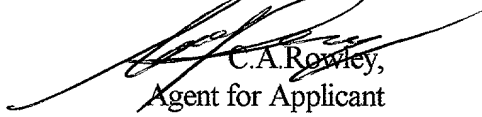
Please file the enclosed patent application and register the enclosed assignment: enclosed are

1. The patent application with abstract, drawings and Declaration and Power of Attorney (signed in 2 parts with only the signature portion of one provided as the remainder is identical to what appears before in the attached form but with the signatures blank.).
2. An Assignment transferring rights to the invention to The University of Western Ontario(signed in 2 parts)..
3. A claim for Small Inventor Status executed by the inventors (signed in 2 parts).
4. A claim for Small Entity Status executed by an Officer of the assignor(signed in 2 parts).

Please charge the Filing (Fee code 201 - \$345.00) and Assignment Fees (Fee Code 581 - \$40.00)(including any additional fees that may apply) to my account no. **18 2150.**

Please return the enclosed post card to provide the Serial No. and filing date for the above.

Respectfully Submitted,


C.A. Rowley,
Agent for Applicant
Registration No. 20,781

Applicant or Patentee: Amarjeet Singh Bassi, Jingxu Zhu, Qing Dao Lan, Argyrios Margaritis, and Ying Zheng

Serial or Patent No.:

Filed or Issued:

For: **Continuous Ion Exchange using a Liquid-Solid Circulating Fluidized Bed**
VERIFIED STATEMENT (DECLARATION) CLAIMING SMALL ENTITY STATUS
(37 CFR 1.9 (f) and 1.27 (D)) - NONPROFIT ORGANIZATION

I hereby declare that I am an official empowered to act on behalf of the nonprofit organization identified below

NAME OF ORGANIZATION **The University of Western Ontario**
ADDRESS OF ORGANIZATION **Room 319, Stevenson-Lawson Building**
London, Ontario, Canada N6A 5B8

TYPE OF ORGANIZATION ☒ University or other institute of higher education

I hereby declare that the nonprofit organization identified above qualifies as a nonprofit organization as defined in 37 CFR 1.9 (e) for purposes of paying reduced fees under section 41 (a) and (b) of Title 35, United States Code with regard to the invention entitled **Continuous Ion Exchange using a Liquid-Solid Circulating Fluidized Bed** inventors **Amarjeet Singh Bassi, Jingxu Zhu, Qing Dao Lan, Argyrios Margaritis, and Ying Zheng** described in

☒ the specification filed herewith
☐ application serial no. _____, filed _____

I hereby declare that the rights under contract or law have been conveyed to and remain with the nonprofit organization with regard to the above identified invention.


I acknowledge the duty to file, in this application or patent, notification of any Change in status resulting in loss of entitlement to small entity status prior to paying, or at the time of paying, the earliest of the issue fee or any maintenance fee due after the date on which status as a small entity is no longer appropriate. (37 CFR 1.28 (b)).

I hereby declare that all the statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements are made with the knowledge that wilful false statements and the like so made are punishable by fine or imprisonment, or both under section 1001 of Title 18 of the United States Code, and that such wilful false statements may jeopardize the validity of the application, any patent issuing thereon, or any patent to which the verified statement is directed.

NAME OF PERSON SIGNING Susan Hoddinott

Title of Person signing Director, Office of Research Services

Address of Person Signing SLB-328, London, Ontario, Canada N6A 5B8

 Signature Susan Hoddinott Date Sept 26 2000.

Applicant or Patentee: **Amarjeet Singh Bassi, Jingxu Zhu, Qing Dao Lan, Argirios Margaritis, Ying Zheng**

Serial or Patent No.:

Filed or Issued:

For: **Continuous Ion Exchange using a Liquid-Solid Circulating Fluidized Bed**
VERIFIED STATEMENT (DECLARATION) CLAIMING SMALL ENTITY STATUS
(37 CFR 1.9 (f) and 1.27 (b)) - INDEPENDENT INVENTOR

As a below named inventor, I hereby declare that I qualify as an independent inventor as defined in 37 CFR 1.9 (c) for purposes of paying reduced fees under section 41 (a) and (b) of Title 35, United States Code to the Patent and Trademark Office with regard to the invention entitled:

Continuous Ion Exchange using a Liquid-Solid Circulating Fluidized Bed

described in

☒ the specification filed herewith

☐ Application Serial No.

☐ Patent no. issued

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Each person, concern or organization to which I have assigned, granted, conveyed or licensed or am under an obligation under contract or law to assign, grant, convey or license any right in the invention is listed below:

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Amarjeet Singh Bassi
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Signature of Inventor

Sept 26, 00
Date

Jinxu Zhu
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Sept. 26, 2000
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Sept 26, 2000
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Ying Zheng
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Date

***NOTE: Separate verified statements are required for each named person, concern or organization having rights to the invention averring to their status as small entities. (37 CFR 1.27)**

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Applicant or Patentee: **Amarjeet Singh Bassi, Jingxu Zhu, Qing Dao Lan, Argirios Margaritis, Ying Zheng**

Serial or Patent No.:

Filed or Issued:

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Continuous Ion Exchange using a Liquid-Solid Circulating Fluidized Bed

described in

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Case	Age	Sex	Duration	Site	Histology	Immunohistochemistry	Molecular biology	Outcome	Comments
1	45	M	10 years	Rectum	Adenocarcinoma	CK20+, CK7+	HER2/neu+	CR	Long-term survival
2	62	F	5 years	Colon	Adenocarcinoma	CK20+, CK7+	HER2/neu-	CR	Long-term survival
3	58	M	8 years	Rectum	Adenocarcinoma	CK20+, CK7+	HER2/neu+	CR	Long-term survival
4	71	F	12 years	Colon	Adenocarcinoma	CK20+, CK7+	HER2/neu-	CR	Long-term survival
5	65	M	7 years	Rectum	Adenocarcinoma	CK20+, CK7+	HER2/neu+	CR	Long-term survival
6	55	F	9 years	Colon	Adenocarcinoma	CK20+, CK7+	HER2/neu-	CR	Long-term survival
7	68	M	6 years	Rectum	Adenocarcinoma	CK20+, CK7+	HER2/neu+	CR	Long-term survival
8	73	F	11 years	Colon	Adenocarcinoma	CK20+, CK7+	HER2/neu-	CR	Long-term survival
9	60	M	10 years	Rectum	Adenocarcinoma	CK20+, CK7+	HER2/neu+	CR	Long-term survival
10	52	F	8 years	Colon	Adenocarcinoma	CK20+, CK7+	HER2/neu-	CR	Long-term survival

***NOTE: Separate verified statements are required for each named person, concern or organization having rights to the invention averring to their status as small entities. (37 CFR 1.27)**

Liquid-Solids Circulation Fluidized Bed

Field of the Invention

The present invention relates to a fluidized bed, more specifically, a liquid-solids circulating fluidized bed arrangement specially suited for ion exchange processes.

5 Background to the Invention

Fluidized beds have been used for a number of different applications such as gas-liquid, gas- solid-liquid, solid-contactors and to carry out a variety of different processes as chemical reactors.

Fluidized beds have found application in ion exchange process. For example Chase, H.A.,
10 "Purification of Proteins by Adsorption Chromatography in Expanded Beds", TIBTECH
12, 296-303 (1994) describes a batch ion exchange process using a conventional fluidized
bed for recovering proteins from whole fermentation broth with the presence of bacterial
cells. It eliminates the difficult solids separation step and recovers the desired products
directly from unclarified whole broth. This process is a batch process employing a
15 conventional fluidized bed.

Burns, M.A. and D.J. Graves, "Continuous Affinity Chromatography Using a Magnetically
Stabilized Fluidized Bed", Biotechnology Progress 1, 95 – 103 (1995) suggested a two-
column magnetically stabilized fluidized bed system for the continuous chromatography of
biochemical products. The magnetically stabilized fluidized bed system is considered to be
20 complicated and costly.

Gordon, N.F., H. Tsujimura and C.L. Cooney, "Optimization and Simulation of
Continuous Affinity Recycle Extraction", Bioseparation 1, 9-12 (1990) describes a process
using mixed reactors as opposed to fluidized bed and reported the continuous affinity
recycle extraction of proteins using well-mixed reactors. This system, although simple and
25 easy to control, has the disadvantage of a stirred tank system – the ion exchange efficiency
is low and large processing volumes are essential for even a moderate throughput
requirement.

Porter and Robert[, US patent 3,879,287: Continuous ion exchange process and apparatus
(1975) relates to an apparatus for continuous ion exchange. However, the process described
30 is a semi-continuous process as the recommended eluting means is a batch wise
conventional fixed bed ion exchange process.

Himsley and Alexander, US patent 4,279,755: Continuous countercurrent ion exchange
process (1993) teaches a continuous countercurrent ion exchange process for absorbing

ions of interest onto ion exchange particles from a feed liquor containing ions which when absorbed on the particles cause the density of the particles to increase. The process comprises the steps of (1) flowing the feed liquor upwardly through a main bed of ion exchange resin particles contained in a main chamber of an absorption column and thereby
 5 maintaining the bed in fluidized state; (2) continuously collecting the denser loaded particles from the lower region of the absorption column; (3) passing an outflow of the feed liquor from the upper region of the main chamber upwardly into the lower region of the polishing chamber containing a secondary bed of fluidized ion exchange resin particles whereby residual ions of interest are polished from the liquor, and (4) producing a barren
 10 liquor flowing out of the upper region of the polishing chamber. Again, this is a semi-continuous process as the stripping and the regeneration of the loaded ion exchange particles cannot be performed in this device.

Brief Description of the Present Invention

It is an object of the present invention to provide a circulating fluidized bed system for
 15 liquid solids contact and interaction, more specifically a Liquid-Solids Circulating Fluidized Bed (LSCFB) ion exchanger.

It is also an object of the present invention to provide a process for continuous recovery of the ions of interest for example contamininants in liquid streams or value added products from waste streams using a Liquid-Solids Circulation Fluidized Bed (LSCFB) ion
 20 exchange system.

Broadly the present invention relates to a fluidized bed system comprising a first fluidized bed, means to feed solids into said first fluidized bed adjacent to a first end of said first fluidized bed and means to feed fluid into said first fluidized bed adjacent to a second end of said first fluidized bed, said second end being remote
 25 from said first end so that said solids and said fluid flow in counter current, a second fluidized bed said second fluidized bed being an entraining fluidized bed wherein a means for introducing solids and a means for introducing fluid into said second bed are both adjacent to the one end of said second fluidized bed so that said solids and said fluid introduced into said second bed flow concurrently through said
 30 second bed from said one end toward another end of said second fluidized bed remote from said one end, first means connecting said first fluidized bed to said second fluidized bed adjacent to said second end of said first fluidized bed and said one end of said second fluidized bed and second means connecting said first and

said second fluidized beds adjacent said first end of said first bed and said other end of said second fluidized bed, said first means connecting being adapted to form a hydraulic seal between said first and second fluidized beds and said second means connecting including said means to feed solids into said first fluidized bed.

- 5 Preferably said first and second fluidized beds are substantially vertical columns. Preferably said second means connecting said first and said second fluidized beds includes a separator means for separating solids from fluid and exhausting such separated fluid to provide separated solids.

- 10 Preferably second means connecting said first and said second fluidized beds further includes a washer for washing said solids before they are feed into said first end of said first fluidized bed.

Preferably said first means connecting said first and said second fluidized beds includes a second washer for washing solids adjacent to said second end of said first fluidized before they are introduced into said second fluidized bed.

- 15 Preferably said first fluidized bed is an absorber for separating ionic products of interest and said second fluidized bed is a desorber for desorption of ionic products and said solids are ion exchange particles. said second means for connecting including said means to feed solids into said first fluidized bed,

- 20 Broadly the present invention also relates to a method or recovering ionic products of interest comprising passing ion exchange particles in countercurrent flow with a feed stream of a first fluid through a first fluidized bed for adsorption of ionic products of interest from said feed stream of said first fluid, transferring said particles with adsorbed ionic products of interest from said first fluidized bed to a second fluidized bed and passing said ion exchange particles with absorbed ionic products in co current flow with an extract buffer of a second fluid through said
25 second fluidized bed for desorption of said adsorbed ionic products of interest, separating said second fluid containing said ionic products of interest desorbed from said ion exchange particles by said second fluid to provide regenerated ion exchange particles and returning said regenerated ion exchanged particles into said
30 first fluidized bed to flow in countercurrent with said first fluid.

Preferably said ion exchange particles with absorbed ionic products are washed before being introduced into said second fluidized bed.

Preferably said ionic product is a protein and said first fluid is a fermentation broth.

Preferably said ionic product is a metal and said first fluid is seawater.

- 5 Preferably said ionic product is an enzyme and said first fluid is dextrose syrup.

Brief Description of the Drawings

Further features, objects and advantages will be evident from the following detailed description of the preferred embodiments of the present invention taken in conjunction with the accompanying drawings in which:

- 10 Figure 1 is a schematic illustration of the method and apparatus of the present invention

Description of the preferred embodiments

Referring to Figure 1 the present invention is composed of a pair of fluidized beds a first fluidized bed 10 and a second fluidized bed 12 interconnected at their adjacent ends by solid transfer and washing systems generally indicated at 14 and 16 respectively. The first fluidized bed 10 is a conventional counter-current flow bed wherein solids (solid particles such as ion exchange beads) as indicated at 18 enter adjacent to the top of the bed 10 as indicated by the line 17 and flow downward and a first fluidizing fluid namely the feed liquor 20 enters the bed 10 as indicated schematically at 22 at the lower end 24 of the bed 10 and flows upward in counter current with the particles 18.

- 20 The second fluidized bed 12 on the other hand is a riser fluidized bed wherein the solid particles 18 transferred from bed 10 via transfer system 14 enter the bed 12 adjacent to the lower end 26 of the bed 12 and flow upward in co-current relation with a second fluidizing fluid 28 (such as extract buffer) which enters the bed 12 under pressure in the illustrated arrangement via nozzle 30 and inlet 32 both adjacent to the lower end 26 of the bed 12 and
25 flows upward through the bed 12 carrying the particles 18 in its flow.

The distributor of the second fluidized bed 12 divides the incoming stream of extracting buffer 28 into two sub-streams: the primary 60 and the auxiliary 62 streams. The primary stream 60 is introduced through nozzle 30 which projects into the second fluidized bed column 12. This design increased the pressure drop across the bottom solids return pipe 42 and makes the system more stable. The auxiliary stream 62 is introduced into the bottom 26 of the second fluidized bed 12 through a perforated plate inlet 32. The function of the auxiliary stream 62 is to stir up the particles at the bottom of the second fluidized bed 12 to be entrained up the second fluidized bed by the combination of the primary and auxiliary

liquid streams 60 and 62. The two streams 60 and 62 may also be combined into a single stream and the fed through a single distributor at the second fluidized bed 12 bottom end 26.

As above indicated the solid particles 18 enter at inlet 17 and travel downward through the bed 10. After they have traversed the fluidized bed 10 the particles 18 enter into the transfer system 14 which includes a washing stage 34 in a conical or funnel shaped bottom end 35 of the housing containing the bed 10 and into which wash water from a source is injected via nozzle 38 positioned adjacent to the apex of the cone in the bottom outlet 40 of the bed 10. The injected wash water 36 travels in counter current to and washes the particles 18 when they leave the fluidized bed 10. The wash water dilutes the feed stream and exits from the top of bed 10 through outlet 44. The washed particles 18 then pass via transfer pipe 42 and are introduced into the second fluidized bed 12.

The function of the wash section 34 is to rinse the loaded particles 18 and to prevent the feed stream 20 from being carried to the second fluidized bed 12 by the particles 18. The bottom solids return pipe 42 is located below the wash section 34. It connects the bottoms of the first fluidized bed 10 and the second fluidized bed 12. During operations, loaded ion exchange particles are transported into the base of the second fluidized bed 12 through the bottom solids return pipe 42 to make up the particles 18 entrained up along the second fluidized bed 12. The bottom solids return pipe 42 operates as a packed moving bed. This is the most important mechanism for forming the dynamic seal between the second fluidized bed 12 and the first fluidized bed 10. The dynamic seal is critical for the success of this continuous ion exchange process, which employs two liquid streams of different properties. In the LSCFB ion exchange system of the present invention, the solids circulation rate is controlled by a butterfly valve schematically indicated at 70 located on the bottom solids return pipe 42. The mechanical valve is preferred over a hydraulic valve due to the low density of the most ion exchange particles, which makes the operation of the hydraulic valve more difficult. An additional advantage of using the mechanical valve in this situation is that it enhances the pressure drop across the solids return pipe 42 and therefore makes the system more stable. The auxiliary liquid stream 62 may be used to provide additional control of the solids circulation rate.

The feed liquor 20 as above described enters at the bottom of the bed 10 travels in countercurrent to the particles 18 through the bed 12 and leave at the top of the bed as

indicated at 44. The fluid exiting from 44 is discarded as waste or as a purified stream in the case of contaminant removal.

The second fluidizing fluid (extract buffer) 28 and the particles 18 from line 42 travel in co- current fashion upward through the bed 12 and are regenerated and then enter the transfer system 16 which includes a separator such as the fluid vortex type separator 46 having a fluid outlet 48 through which the second fluidizing fluid 28 is removed and a solids outlet through a washing stage 50 at the bottom. This fluid exiting from outlet 48 contains the ions of interest and may be subjected to further downstream processing or membrane treatment to concentrate the ions of interest. Washing fluid is injected via nozzle 52 at the bottom of the washing stage 50 and flow upward in countercurrent with the downcoming solids (regenerated solid particles) 18 and the so washed particles 18 enter the inlet tube delivering the regenerated particles 18 into the top of the bed 10. The washing fluid dilutes the extract buffer and exits from the outlet 48.

The operation of the invention will be described in relation to ion exchange process, but it may be used in other potential application as described below.

In the process of ion exchange, the feed liquor 20 is introduced via inlet 22 into the bottom (second) end of the first fluidized bed 10 (downcomer 10) and the regenerated particles 18 from the bed 12 are introduced via line 17 adjacent to the first or the top of the first fluidized bed 10 i.e. the feed 20 and regenerated beads are introduced at opposite ends of the first fluidized bed 10.

The falling particles 18 and the up-flowing feed liquor 20 contact counter-currently and the target ions in the feed 20 are adsorbed onto the ion exchange particles 18 in the first fluidized bed 10. The de-ionized liquor leaves from the top of the first fluidized bed through the raffinate outlet 44 and the loaded particles 18 fall into the washing stage 34 at the base of the first fluidized bed 10 are rinsed and then transferred via line 42 to the base of the second fluidized bed 12.

During operations, as above described loaded ion exchange particles are transported into the base of the second fluidized bed 12 through the bottom solids return pipe 42 to make up the particles 18 entrained up along the second fluidized bed 12. The bottom solids return pipe 42 operates as a packed moving bed forming the dynamic seal between the second fluidized bed 12 and the first fluidized bed 10.

The extracting buffer 28 is applied to the second fluidized bed 12 at the bottom. The superficial liquid velocity in the second fluidized bed 12 is maintained in a range higher

than the terminal velocity of the ion exchange particles 18 so that the loaded particles are carried upward by the upflowing buffer 28. The buffer 28 and the loaded ion exchange particles 18 hence contact co-currently while desorption and regeneration of the particles 18 proceed in the second fluidized bed 12. The extract 28 and the regenerated ion exchange particles 18 are separated by a liquid-solids separator 46 adjacent to the top of the second fluidized bed 12. The extract is then collected from the extract outlet 48 and the regenerated ion exchange particles 18 returned to the first fluidized bed 10 through the top solids return pipe 17, after being rinsed through the wash section 50.

The liquid-solids separator 46 in the illustrated arrangement is a hydraulic (but can be any other type of separator) cyclone, which separates the regenerated particles 18 from the extract 28. The extract outlet 48 is located on the separator preferably at the same level as that of the raffinate outlet 44 on the top of the first fluidized bed 10 to maintain the pressure balance between the second fluidized bed 12 and the first fluidized bed 10. To prevent the loss of particles through the extract outlet, a stainless steel mesh (not shown) is preferably used to cover the extract outlet 48.

The top washing section 50 comprises of the funnel bottom of the separator and return pipe 17. The upward washing water slows down the falling of the particles 18 and creates a solids layer in the funnel bottom of the separator 46. It also rinses the particles 18 before their falling into the top solids return pipe 17 and minimizes the inter-mixing between the extract in the second fluidized bed 12 and the de-ionized liquor at the top of the first fluidized bed 10. The return pipe 17 (particle inlet to the first fluidized bed 10) enters the first fluidized bed 10 sufficiently below the outlet 44 to maintain a freeboard section 64 in the upper part of the first fluidized bed 10 of sufficient height to substantially eliminate carry over of particles 18 through the outlet 44.

25 **Applications of the present invention**

A feed liquor 20 from which ions can be recovered, such as a fermentation broth, usually contains a large amount of small solids and relatively low concentration of desired product(s). Hence, the first task in developing a new downstream treatment process usually focuses on the selection of an appropriate procedure for handling the solids present in the feed. This is typically achieved by filtration or centrifugation. However, the presence of colloidal solids and the viscous properties of many feeds frequently make those methods both costly and inefficient. The LSCFB ion exchange system of the present invention is an

integrated unit operation which can recover desired ions from unclarified whole broth continuously.

The desorption of the target ions and the regeneration of the ion exchange particles are carried out in the second fluidized bed 12. The loaded ion exchange particles 18 are transported into the base of the second fluidized bed 12 through the bottom solids return system 14 and flow co-currently upward with the extracting buffer 28 along the second fluidized bed 12. The loaded particles are stripped of the target ions and regenerated in the second fluidized bed 12 before being entrained into the liquid-solids separator 46 of the transfer system 16. As the second fluidized bed 12 is operated in the circulating fluidization regime with high liquid velocity, the contact efficiency and the mass transfer rate between the liquid and solids are very high.

In the liquid solids circulation fluidized bed (LSCFB), diagrammed in Figure 1, the adsorption in the first fluidized bed or downcomer 10 and the desorption in the second fluidized bed or second fluidized bed 12 can be carried out in a continuous mode with the ion exchange particles circulated continuously between the two columns. The ion exchange particles 18 employed in this system should have reasonably large adsorption capacity to the target or desired ions and the density of the ion exchange particles 18 in the swollen state should be larger than that of the feed liquor. As the first fluidized bed 10 is maintained in the conventional fluidization regime, the bed voidage could be adjusted to allow the passage of the particulates in an unclarified feed by controlling the superficial liquid velocity in the first fluidized bed. In other words, this system can be used to purify the target ions directly from an unclarified whole broth so that the costly pre-clarification process is eliminated.

In the LSCFB, the adsorption of the target ions are carried out in the first fluidized bed 10 and the desorption and the regeneration in the second fluidized bed 12. This is a continuous process with the ion exchange particles 18 circulated continuously between the two columns 10 and 12. Two different liquid streams, the feed 20 in the first fluidized bed 10 and the extracting buffer 28 in the second fluidized bed 12, are used in this system. The second fluidized bed 12 is operated in the circulating fluidization regime and the first fluidized bed in the conventional fluidization regime.

Examples

In an arrangement as shown in Figure 1, the second fluidized bed 12 is an acrylic column of I.D. 38.1 mm and 3 m in height. The distributor of the second fluidized bed 12 divides

the incoming stream of extracting buffer is divided into two substreams: the primary 60 and the auxiliary 62 streams. The primary stream 60 is introduced through a stainless steel pipe (I.D. 11 mm) (nozzle 30). It projects 36 mm into the second fluidized bed column 12. Since the liquid velocity in the second fluidized bed is maintained in a range higher than the terminal velocity of the ion exchange particles, the high liquid velocity enhances the contact efficiency and also the mass transfer rate between the liquid and the particles.

The top washing section 50 as above described comprises of the funnel bottom of the separator 46 and an acrylic pipe of 40 mm in diameter and 200 mm in height (pipe 17).

The first fluidized bed is a Plexiglas column of I.D. 120 mm and 2.5 m in height. The particle entrance 17 on the first fluidized bed 10 is located 0.813 m below the raffinate outlet 44 to prevent the direct loss of particles through the raffinate outlet 44. The distributor 22 of the first fluidized bed 10 is a perforated stainless steel pipe. This distributor allows the particles to fall through to the bottom solids return pipe 42 while introducing the feed 20 to the first fluidized bed 10.

The bottom washing section 34 is comprised of the funnel bottom of the first fluidized bed 10 and a vertical pipe 40 of 40 mm I.D. and 200 mm in height. Wash water is introduced from the base of this column and goes upward (nozzle 38).

In the LSCFB ion exchange system, the solids circulation rate is controlled as above described by a butterfly valve 70 located on the bottom solids return pipe 42.

Table 1 summarizes the experimental result conducted using the apparatus as above described, with whole whey which contains approximately 5.4 g/L proteins and with an artificial protein solution, the 2 g/L bovine albumin serum (BSA) solution. The protein recovery from BSA solution was much higher than that from the whey solution. This is because the high ionic strength and the fouling effects of the milk-fats in whey solution reduced the dynamic capacity of the system.

Table 1. Summary of parameters of whey protein recovery under different conditions

Feed Type	Protein Conc. in Feed (g/L)	Feed Flow rate (L/hr)	Protein Loading Rate (g/hr)	Protein Conc. in Raffinate (waste feed) (g/L)	Overall Recovery (%)	Throughput (g/hr·(kg beads))
Whey	5.4	5.7	31.2	0.77	78.4	8.2
BSA Solution	2.0	38.4	76.8	0.79	84.0	21.5

Potential Technology Applications

- 5 Potential applications of the invention that the invention is believed to be suitable for include:
- a) The recovery of ionic products from biological or non-biological feeds such as protein recovery from fermentation broth, metal recovery from sea water, etc. where suitable ion exchange particles are available;
 - 10 b) The removal of ionic contaminants from products or intermediate products, e.g., removal of enzyme from dextrose syrup after the conversion;
 - c) The desalination of water;
 - d) Wastewater treatment.

In Summary

- 15 **Ion Exchange of Target Ions occurs by:**
1. Regenerated ion exchange particles are fed to the first fluidized bed through the top solids return pipe; those particles flow down to the lower part of the first fluidized bed to form a particulate bed;
 2. The feed liquor flows upward through the down moving bed of ion exchange particles and maintains the bed in the conventional fluidized regime;
 - 20 3. The target ions are adsorbed onto the ion exchange particles when the ion exchange particles and the feed contact counter-currently in the particulate bed;
 4. The de-ionized liquid is discarded from the raffinate outlet and the loaded ion exchange particles fall into the bottom wash section;
 - 25 5. The rinsed ion exchange particles are continuously transported to the second fluidized bed through the bottom solids return pipe;

6. Extracting buffer is fed into the base of the second fluidized bed and flows upward at a velocity higher than the terminal velocity of the particles, thereby maintained in a circulating fluidization regime;
7. The loaded particles are desorbed and regenerated while being entrained up continuously along the second fluidized bed;
8. The regenerated particles are separated from the extract in the liquid-solids separator at the top; the extract is collected from the extract outlet on the liquid-solid separator and the regenerated particles are rinsed in a wash section below the separator;
9. The rinsed particles are fed to the first fluidized bed by gravity. Another cycle begins.

Having described the invention, modifications will be evident to those skilled in the art without departing from the spirit of the invention as defined in the appended claims.

Claims

1. A fluidized bed system comprising a first fluidized bed, means to feed solids into said first fluidized bed adjacent to a first end of said first fluidized bed and means to feed fluid into said first fluidized bed adjacent to a second end of said first fluidized bed, said second end being remote from said first end so that said solids and said fluid flow in counter current, a second fluidized bed, said second fluidized bed being an entraining fluidized bed wherein a means for introducing solids and a means for introducing fluid into said second bed are both adjacent to the one end of said second fluidized bed so that said solids and said fluid introduced into said second bed flow concurrently through said second bed from said one end toward another end of said second fluidized bed remote from said one end, first means connecting said first fluidized bed to said second fluidized bed adjacent to said second end of said first fluidized bed and said one end of said second fluidized bed and second means connecting said first and said second fluidized beds adjacent said first end of said first bed and said other end of said second fluidized bed, said first means connecting being adapted to form a hydraulic seal between said first and second fluidized beds and said second means connecting includes said means to feed solids into said first fluidized bed.
2. A fluidized bed system as defined in claim 1 wherein said first and second fluidized beds are substantially vertical columns.
3. A fluidized bed system as defined in claim 2 wherein said second means connecting said first and said second fluidized beds includes a separator means for separating solids from fluid and exhausting such separated fluid to provide separated solids
4. A fluidized bed system as defined in claim 3 wherein second means connecting said first and said second fluidized beds further includes a washer for washing said solids before they are fed into said first end of said first fluidized bed.
5. A fluidized bed system as defined in claim 2 wherein said first means connecting said first and said second fluidized beds includes a second

washer for washing solids adjacent to said second end of said first fluidized before they are introduced into said second fluidized bed.

6. A fluidized bed system as defined in claim 3 wherein said first means connecting said first and said second fluidized beds includes a second washer for washing solids adjacent to said second end of said first fluidized before they are introduced into said second fluidized bed.
7. A fluidized bed system as defined in claim 4 wherein said first means connecting said first and said second fluidized beds includes a second washer for washing solids adjacent to said second end of said first fluidized before they are introduced into said second fluidized bed.
8. A fluidized bed system as defined in claim 1 wherein said first fluidized bed is an absorber for separating ionic products of interest and said second fluidized bed is a desorber for desorption of ionic products and said solids are ion exchange particles. said second means for connecting including said means to feed solids into said first fluidized bed.
9. A fluidized bed system as defined in claim 2 wherein said first fluidized bed is an absorber for separating ionic products of interest and said second fluidized bed is a desorber for desorption of ionic products and said solids are ion exchange particles. said second means for connecting including said means to feed solids into said first fluidized bed.
10. A fluidized bed system as defined in claim 3 wherein said first fluidized bed is an absorber for separating ionic products of interest and said second fluidized bed is a desorber for desorption of ionic products and said solids are ion exchange particles. said second means for connecting including said means to feed solids into said first fluidized bed.
11. A fluidized bed system as defined in claim 4 wherein said first fluidized bed is an absorber for separating ionic products of interest and said second fluidized bed is a desorber for desorption of ionic products and said solids are ion exchange particles. said second means for connecting including said means to feed solids into said first fluidized bed.
12. A fluidized bed system as defined in claim 5 wherein said first fluidized bed is an absorber for separating ionic products of interest and said second fluidized bed is a desorber for desorption of ionic products and said solids

are ion exchange particles. said second means for connecting including said means to feed solids into said first fluidized bed.

13. A fluidized bed system as defined in claim 6 wherein said first fluidized bed is an absorber for separating ionic products of interest and said second fluidized bed is a desorber for desorption of ionic products and said solids are ion exchange particles. said second means for connecting including said means to feed solids into said first fluidized bed.

14. A fluidized bed system as defined in claim 7 wherein said first fluidized bed is an absorber for separating ionic products of interest and said second fluidized bed is a desorber for desorption of ionic products and said solids are ion exchange particles. said second means for connecting including said means to feed solids into said first fluidized bed.

15. A method for recovering ionic products of interest comprising passing ion exchange particles in countercurrent flow with a feed stream of a first fluid through a first fluidized bed for adsorption of ionic products of interest from said feed stream of said first fluid, transferring said particles with adsorbed ionic products of interest from said first fluidized bed to a second fluidized bed and passing said ion exchange particles with adsorbed ionic products in co current flow with an extract buffer of a second fluid through said second fluidized bed for desorption of said adsorbed ionic products of interest, separating said second fluid containing said ionic products of interest desorbed from said ion exchange particles by said second fluid to provide regenerated ion exchange particles and returning said regenerated ion exchanged particles into said first fluidized bed to flow in countercurrent with said first fluid.

16. A method as defined in claim 15 wherein said ion exchange particles with adsorbed ionic products are washed before being introduced into said second fluidized bed

17. A method as defined in claim 16 wherein said regenerated ion exchange particles are washed before being returned to said first fluidized bed

18. A method as defined in claim 16 wherein said ionic product is a protein and said first fluid is a fermentation broth.

19. A method as defined in claim 16 wherein said ionic product is a metal and said first fluid is sea water.
20. A method as defined in claim 16 wherein said ionic product is an enzyme and said first fluid is dextrose syrup.

Abstract of the Disclosure**Liquid-Solids Circulation Fluidized Bed**

A continuous Liquid-Solids Circulating Fluidized Bed (LSCFB) preferably for use as an
5 ion exchanger consists of two fluidized bed columns, a fluidized bed adsorber (downer)
operating in conventional fluidized bed mode for adsorption of ions of interest and a
fluidized bed riser for desorption of ions (operating as a riser fluidized bed) to provide
regenerated particles. Ion exchange particles circulate continuously between the riser and
the downer i.e. the particles that have adsorbed ions in the adsorber pass from the adsorber
10 (downer) to the desorber where they are regenerated and the so regenerated particles are
return to the adsorber near the top of the adsorber column. The LSCFB can be used in
processes for continuous recovery of the ions of interest.

The diagram illustrates a continuous extraction system. It features two vertical columns. The left column has an inlet at the top (16) and an outlet at the bottom (30). The right column has an inlet at the top (48) and an outlet at the bottom (38). A hopper (46) feeds material into the top of the right column. A series of horizontal baffles or trays are positioned within both columns, labeled 17, 18, and 18. Arrows indicate the flow direction: downwards in the right column and upwards in the left column. At the bottom of the right column, there is a collection point (24) leading to a container (20) labeled "Feed Liquor". Another line from the bottom of the right column leads to a container (36) labeled "Washing Water". The bottom of the left column is connected to a container (28) labeled "Extract Buffer" via a line (32). Various other components are labeled with numbers: 10, 12, 14, 22, 26, 34, 35, 40, 42, 44, 50, 52, 60, 62, 64, 70.

Figure 1

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I PTO/SB/01 (3-97)

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DECLARATION FOR UTILITY OR DESIGN PATENT APPLICATION	Attorney Docket Number	UWO3		
	First Named Inventor	Amarjeet Singh Bassi		
	COMPLETE IF KNOWN			
	Application Number			
	Filing Date			
	Group Art Unit			
<input checked="" type="checkbox"/> Declaration Submitted with Initial Filing	OR	<input type="checkbox"/> Declaration Submitted After Initial Filing	Examiner Name	

As a below named inventor, I hereby declare that:

My residence, post office address, and citizenship are as stated below next to my name.

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

Continuous Ion Exchange using a Liquid-Solid Circulating Fluidized Bed

(Title of the Invention)

the specification of which

☒ is attached hereto

OR

☐ was filed on (MM/DD/YYYY) as United States Application No. or PCT International Application No.

and was amended on (MM/DD/YYYY) (if applicable)

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment specifically referred to above.

I acknowledge the duty to disclose information which is material to patentability as defined in Title 37 Code of Federal Regulations §1.56.

I hereby claim foreign priority benefits under Title 35, United States Code §119(a)-(d) or §365(b) of any foreign application(s) for patent or inventor's certificate, or §365(a) of any PCT international application which designated at least one country other than the United States of America, listed below and have also identified below, by checking the box, any foreign application for patent or inventor's certificate, or of any PCT international application having a filing date before that of the application on which priority is claimed.

Prior Foreign Application Numbers	Country	Foreign Filing Date (MM/DD/YYYY)	Priority Not Claimed	Certified Copy Attached?	
				YES	NO
			<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
			<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
			<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
			<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
			<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
			<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>

Additional foreign application nos. are listed on a supplemental priority data sheet PTO/SB/02B attached hereto.

I hereby claim the benefit under Title 35, United States Code §119(e) of any United States provisional application(s) listed below:

Application Number(s)	Filing Date (MM/DD/YYYY)	Additional provisional application numbers are listed on a supplemental priority data sheet PTO/SB/02B attached hereto.

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DECLARATION - Utility or Design Patent Application

I hereby claim the benefit under Title 35, United States Code §120 of any United States application(s), or §366© of any PCT International application designating the United States of America, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT International application in the manner provided by the first paragraph of Title 35, United States Code §112, I acknowledge the duty to disclose information which is material to patentability as defined in Title 37, code of Federal Regulations §1.58 which became available between the filing date of the prior application and the national or PCT International filing date of this application.

U.S. Parent Application Number	PCT Parent Number	Parent Filing Date (MM/DD/YYYY)	Parent Patent Number (if applicable)

☐ Additional US or PCT International application nos are listed on a supplemental priority data sheet PTO/SB/02B attached hereto

As a named inventor, I hereby appoint the following registered practitioner(s) to prosecute this application and to transact all business in the Patent and Trademark Office connected therewith:

<input type="checkbox"/> Customer Number		Place Customer Number Bar Code Label Here	
OR <input checked="" type="checkbox"/> Registered practitioner(s) name/registration no. listed below			
Name	Registration Number	Name	Registration Number
C.A. Rowley	20,781		

☐ Additional registered practitioner(s) named on supplemental Registered Practitioner Info sheet PTO/SB/02C attached hereto.

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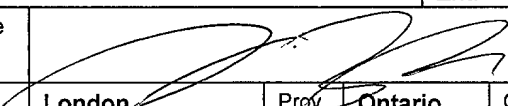
I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Name of Sole or First Inventor: ☐ A petition has been filed for this unsigned inventor
 Given Name (first and middle [if any]) **Amarjeet Singh** Family Name or Surname **Bassi**

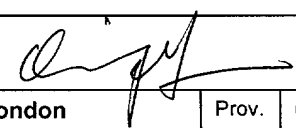
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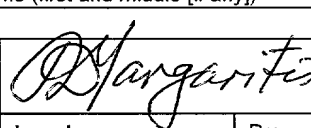
I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

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Name of Fifth Inventor:				A petition has been filed for this unsigned inventor			
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Inventor's Signature		<i>Ying Zheng</i>				Date	
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